GERMAN REICH

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Addition to the Patent 557 428

Patented in the German Reich from 25 December 1930

The main patent commenced on 20 December 1930

In the process of Patent 557 428, sulphuric esters of alcohols are prepared by heating these with salts of imidodisulphonic acid.

It has further been found that heating alcohols with the salts of the acidic sulphuric esters of lower alcohols likewise leads smoothly to the sulphuric ester 10 of the starting alcohol. The lower alcohol formed for example according to the reaction equation

$$C_2 H_5 O S O_3 K + R O H$$

= $R - O S O_3 K + C_2 H_5 O H$

15 is best removed by distillation.

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^{*)} The following have been reported as inventors by the Applicant:
Dr Karl Brodersen and Dr Matthias Quaedvlieg of Dessau

The process thus differs fundamentally from the known introduction of alkyl radicals into other species by means of alkylsulphuric acid, with sulphuric acid or salts thereof being liberated, since in this case, conversely, the sulphuric acid radical is being introduced while alcohol exits.

The reaction ensues at a moderate temperature, but it is better to utilize temperatures of 100° or higher.

10 Catalysts such as soap, for example, make the reaction go quickly.

The process is equally applicable to primary alcohols, secondary alcohols and tertiary alcohols.

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True, it has already become known to replace the glyceryl radical of esters of higher fatty acids with the radical of a primary alcohol, but that amounts to the transesterification of an organic ester. It certainly does not suggest that it is possible for salts of acidic sulphuric esters to take part in a reaction

$$R \cdot OSO_3 Na + R' \cdot OH$$

= $R' \cdot OSO_3 Na + R \cdot OH$

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On the contrary, the expectation was that the alkyl sulphate salts would react with the oxy compounds to form alkyl ethers

$$R \cdot OSO_3 Na + R' \cdot OH$$

= $R \cdot O \cdot R' + Na HSO_4$,

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as is known to be the case in an aqueous medium.

Example 1

35 24 parts of cetyl alcohol are melted and admixed with

20 parts of potassium ethylsulphate by stirring. Stirring is continued while gradually heating to 120-150° and maintaining this temperature until the alcohol distillation ceases. Potassium cetylsulphate is left behind.

Example 2

40 parts of a mixture of primary, secondary and tertiary alcohols of average molecular weight 195, as is obtainable by oxidation of paraffin, are treated with a trace of a sodium soap and 40 parts of potassium ethylsulphate according to Example 1. The potassium salt of the corresponding sulphuric ester is formed.

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Example 3

40 parts of oleyl alcohol and 30 parts of potassium ethylsulphate are thoroughly stirred while being 20 rapidly heated to 160° until the elimination of alcohol, which ensues with lively foaming, has ended. If necessary the white powder staying behind is freed of admixed traces of unsulphonated alcohol by extraction with ether for example. The yield amounts to about 65 parts of potassium oleylsulphate.

CLAIM:

Process for preparing sulphuric esters of alcohols of the aliphatic series, characterized in that, in modification of the process according to Patent 557 428, alcohols are treated with salts of the acidic sulphuric esters of such alcohols as have a lower molecular weight than the alcohols to be esterified.